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[Title of the Invention] Process for Preparing Uni-Core Detergent Particle

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[Document] Specification 1

[Document] Abstract 1

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[Document] Specification

[Title of the Invention] Process for Preparing Uni-Core Detergent Particle

[Claims]

[Claim 1] A process for preparing a uni-core detergent particle, comprising the steps of
(A): mixing a base particle and 20 to 100 parts by weight of a surfactant composition, based on 100 parts by weight of said base particle, the base particle having an average particle size of from 150 to 500 µm, a bulk density of 400 g/L or more, and a particle strength of 50 kg/cm^2 or more, under mixing conditions such that said base particle does not substantially undergo breakdown, to give a mixture; and
(B): mixing the mixture obtained in step (A) with 5 to 100 parts by weight of fine powder, based on 100 parts by weight of the mixture, with substantially maintaining the shape of the base particle containing the surfactant composition, to give a uni-core detergent particle, wherein the uni-core detergent particle has a degree of particle growth of 1.5 or less, and a bulk density of 500 g/L or more.

[Claim 2] The process according to claim 1, wherein in said step (A), a mixing operation is carried out by using a mixer comprising agitation impellers, wherein the

agitation impellers have a Froude number of from 0.5 to 8, provided where the mixer further comprises disintegration impellers, the mixing operation is carried out under mixing conditions so as not to substantially rotate the disintegration impellers.

[Claim 3] The process according to claim 1 or 2, wherein in said step (B), a mixing operation is carried out by using a mixer comprising agitation impellers and disintegration impellers, under mixing conditions that the agitation impellers have a Froude number of 2 or more, and that the disintegration impellers have a Froude number of 200 or more.

[Claim 4] The process according to any one of claims 1 to 3, wherein the mixture obtainable in step (A) has any one of forms in Funicular II region, Capillary region, and Slurry region.

[Claim 5] The process according to any one of claims 1 to 4, wherein said surfactant composition comprises a nonionic surfactant, 0 to 300 parts by weight of an anionic surfactant having sulfate group or sulfo group, based on 100 parts by weight of the nonionic surfactant, and 1 to 100 parts by weight of an immobilization agent for the nonionic surfactant, based on 100 parts by weight of the nonionic surfactant.

[Detailed Description of the Invention]

[0001]

[Technical Field to Which the Invention Pertains]

The present invention relates to a process for preparing a uni-core detergent particle comprising adding a surfactant composition.

[0002]

[Prior Art]

A process in which a liquid surfactant such as a nonionic surfactant is used for a powder detergent includes a process of supporting a liquid surfactant on a powder.

[0003]

Japanese Patent Laid-Open No. Sho 52-110710 discloses a granular detergent comprising a liquid or liquefiable organic substance contained in an inner portion of base material beads having porous outer surface and skeletal inner structure, wherein a nonionic surfactant is not substantially present on the beads surface. However, in this technique, the beads cannot include liquid ingredients in amounts of not less than an oil-absorbable amount, and moreover, liquids are more likely to remain on the particle surface as the amount of the surfactant formulated increases, thereby making its flowability poor. Therefore, the amount of the surfactant formulated by this technique cannot be increased.

[0004]

Japanese Patent Laid-Open No. Hei 5-209200 discloses a process for preparing a nonionic detergent particle, comprising using, as raw materials for detergents, a mixture comprising a nonionic surfactant as a main base material; forming a deposition layer of the raw materials for detergents on a wall of an agitation mixer comprising agitation impellers and having a clearance between the agitation impellers and the mixer wall; and granulating with increasing a bulk density by the agitation impellers. However, in this technique, the process is complicated, and as the amount of a surfactant formulated varies, the particle size of the detergent particle varies. Further, in this technique, as a matter of course, the deposition of the raw materials for detergents in the mixer is remarkable, which may cause variations in the particle size and the bulk density of the detergent particle depending upon the deposition conditions.

[0005]

Therefore, in order to produce products having stable quality, it is very important to construct a process for preparing powder detergents for obtaining products having stable quality with respect to the variation in the amount of the nonionic surfactant formulated. Especially, although a powder detergent having a high liquid

surfactant content is excellent in the deterging performance, it has been difficult to produce a product having stable quality for the reasons given above, and the problem of adversely changing powder properties by containing a large amount thereof has not be solved thereby.

[0006]

[Problems to Be Solved by the Invention]

Accordingly, a problem of the present invention is to provide in a process for preparing a uni-core detergent particle comprising a surfactant composition, the process for obtaining the uni-core detergent particle in high yield capable of easily adjusting an average particle size and a particle size distribution by selection of a base particle in a simple preparation process, wherein the variations of the average particle size and the particle size distribution of the detergent particle are small with respect to the variation of the amount of the surfactant composition formulated. Further, a problem of the present invention is to provide a process for preparing a uni-core detergent particle which is excellent in powder properties, such as flowability, of the detergent particle, and is capable of formulating a large amount of the surfactant composition.

[0007]

[Means to Solve the Problems]

Specifically, the gist of the present invention relates to a process for preparing a uni-core detergent particle, comprising the steps of

(A): mixing a base particle and 20 to 100 parts by weight of a surfactant composition, based on 100 parts by weight of the base particle, the base particle having an average particle size of from 150 to 500 µm, a bulk density of 400 g/L or more, and a particle strength of 50 kg/cm^2 or more, under mixing conditions such that the base particle does not substantially undergo breakdown, to give a mixture; and

(B): mixing the mixture obtained in step (A) with 5 to 100 parts by weight of fine powder, based on 100 parts by weight of the mixture, with substantially maintaining the shape of the base particle containing the surfactant composition, to give a uni-core detergent particle, wherein the uni-core detergent particle has a degree of particle growth of 1.5 or less, and a bulk density of 500 g/L or more.

[0008]

[Modes for Carrying out the Invention]

1. Base Particle

In the present specification, the term "base particle" refers to a particle having an average particle size of from 150 to 500 µm, a bulk density of 400 g/L or

more, and a particle strength of 50 kg/cm^2 or more.

[0009]

The base particle has an average particle size of from 150 to 500 μm, preferably from 180 to 350 μm, from the viewpoint of obtaining a uni-core detergent particle excellent in the dissolubility and the flowability. The bulk density is 400 g/L or more, preferably 500 g/L or more, from the viewpoint of the compression of the detergent particles. Also, the bulk density is preferably 1500 g/L or less, more preferably 1200 g/L or less, from the viewpoint of the dissolubility. The particle strength is 50 kg/cm^2 or more, and from the viewpoint of the stable productivity of the uni-core detergent particle, the particle strength is preferably 100 kg/cm^2 or more, more preferably 200 kg/cm^2 or more. The particle strength is 5000 kg/cm^2 or less, more preferably 3000 kg/cm^2 or less, from the viewpoint of the dissolubility. If a base particle has a particle strength within this range, the breakdown of the base particle during mixing in step (A) is substantially suppressed.

[0010]

The average particle size is measured by vibrating a sample with each of standard sieves according to JIS Z 8801 for 5 minutes, and thereafter determining from a weight percentage depending upon the size openings of

the sieves.

The bulk density is measured by a method according to JIS K 3362.

[0011]

The particle strength is measured by the following method.

A cylindrical vessel of an inner diameter of 3 cm and a height of 8 cm is charged with 20 g of a sample, and the sample-containing vessel is tapped for 30 times. The sample height at this time is measured. Thereafter, an entire upper end surface of the sample kept in the vessel is pressed at a rate of 10 mm/min with a pressing machine to take measurements for a load-displacement curve. The slope of the linear portion at a displacement rate of 5% or less is divided by a product obtained by multiplying an initial sample height and a pressed area, to give a quotient which is defined as particle strength.

[0012]

The base particle in the present invention may be particle of any substances which is generally blended in a detergent and dissolved or dispersed in water. The base particle includes, for example, particle exhibiting alkaline property such as tripolyphosphates, carbonates, bicarbonates, sulfites, silicates, crystalline aluminosilicates, and citrates; particle exhibiting

neutral property or acidic property such as sodium sulfate, sodium chloride, and citric acid; or particle prepared by drying an aqueous slurry comprising various detergent builders by means of spray-drying or the like. The base particle may be constituted by single component alone, or may be constituted by a plurality of components.

[0013]

Among them, the particle prepared by drying an aqueous slurry comprising a detergent builder are preferable as particle, from the viewpoint that the formulated amount of the surfactant composition can be made large. The base particle can be prepared, for example, by spray-drying an aqueous slurry comprising a water-insoluble inorganic compound, a water-soluble polymer and a water-soluble salt, in which the contents of each of the components are respectively from 20 to 90% by weight, from 2 to 30% by weight, and from 5 to 78% by weight, on the basis of solid ingredients in the aqueous slurry. Within the above compositional ranges, the particle strength, the bulk density and the average particle size of the base particle can be controlled by adjusting the drying process and the drying conditions.

[0014]

Here, the water-insoluble inorganic compound includes crystalline or amorphous aluminosilicates; silicon dioxide,

hydrated silicate compounds, clay compounds such as perlite and bentonite, and the like. The water-soluble polymer includes carboxylic acid-based polymers, carboxymethyl cellulose, water-soluble starches, sugars, and the like. The water-soluble salts include water-soluble inorganic salts representatively exemplified by alkali metal salts, ammonium salts or amine salts, each having carbonate radical, hydrogencarbonate radical, sulfate radical, sulfite radical, hydrogensulfate radical, chloride radical, phosphate radical, or the like; and water-soluble organic salts having low molecular weights such as citrates and fumarates, and the like.

[0015]

The contents of the water-insoluble inorganic compound, the water-soluble polymer and the water-soluble salt in the aqueous slurry are respectively more preferably within the ranges of from 30 to 75% by weight, from 3 to 20% by weight, and from 10 to 67% by weight, especially preferably within the ranges of from 40 to 70% by weight, from 5 to 20% by weight, and from 20 to 55% by weight, on the basis of solid ingredients in the aqueous slurry.

[0016]

2. Surfactant Composition

The surfactant composition includes, for instance, a

composition comprising a surfactant exhibiting a liquid state during the mixing operation of step (A). Therefore, in addition to liquid surfactants at the temperature of mixing operation, even a solid surfactant at that temperature can be used in this process as the surfactant, as long as the surfactant can be obtained as a solution or suspension for dissolving or dispersing in an appropriate medium.

[0017]

As the surfactant, an anionic surfactant, a nonionic surfactant, an amphoteric surfactant and a cationic surfactant may be used alone, or in combination of two or more kinds. In addition, in the present specification, one of the embodiments of the surfactant composition includes a surfactant composition comprising a nonionic surfactant; 0 to 300 parts by weight of an anionic surfactant having sulfate group or sulfo group, based on 100 parts by weight of the nonionic surfactant; and 1 to 100 parts by weight of an immobilization agent of the nonionic surfactant, based on 100 parts by weight of the nonionic surfactant. The anionic surfactant having sulfate group or sulfo group is more preferably from 20 to 200 parts by weight, based on 100 parts by weight of the nonionic surfactant. The surfactant composition having the above composition is more preferable, because the

desirable foaming ability and detergency performance can be obtained.

[0018]

In the present specification, the term "immobilization agent" means a base material capable of suppressing the flowability of the nonionic surfactant which is liquid at an ordinary temperature and remarkably enhancing the hardness in a state in which the flowability of the surfactant composition comprising the above nonionic surfactant is lost. The above immobilization agent includes, for instance, anionic surfactants such as salts of fatty acids, salts of hydroxyfatty acids, and alkyl phosphates; polyoxyalkyl-type nonionic compounds such as polyethylene glycols; polyether-type nonionic compounds, and the like. The immobilization agent is more preferably from 5 to 50 parts by weight, based on 100 parts by weight of the nonionic surfactant. The exudation of the surfactant during storage at ordinary temperature can be suppressed, because the surfactant composition comprising an immobilization agent is used.

[0019]

The amount of the surfactant composition formulated is 20 parts by weight or more, preferably 25 parts by weight or more, more preferably 30 parts by weight or more, based on 100 parts by weight of the base particle, from

the viewpoint of exhibiting the detergency. The amount is 100 parts by weight or less, preferably 80 parts by weight or less, more preferably 70 parts by weight or less, based on 100 parts by weight of the base particle, from the viewpoints of the dissolubility and the flowability.

[0020]

3. Powder Raw Materials Other Than the Base Particle

The term "powder raw materials other than the base particle as referred to in the present specification means a detergency-enhancing agent or an oil-absorbing agent which is powdery at an ordinary temperature. Concretely, the powder raw materials are base material agents exhibiting a metal ion capturing ability such as zeolite and citrates; base material agents exhibiting an alkalizing ability such as sodium carbonate and potassium carbonate; base material agents exhibiting both a metal ion capturing ability and an alkalizing ability such as crystalline silicates; amorphous silica and amorphous aluminosilicates exhibiting poor metal ion capturing ability but high oil-absorbing ability, and the like. By using the above powder raw materials in combination with the base particle as desired, an increase in the amount of the surfactant composition formulated and the reduction of the deposition of the mixture within the mixer can be achieved, and the improvement in detergency can also be

achieved.

[0021]

The powder raw materials other than the base particle are mixed by formulating as desired in step (A). The formulation amount, based on 100 parts by weight of the base particle, is preferably 1 part by weight or more, more preferably 3 parts by weights, from the viewpoint of exhibiting given effects. In addition, the formulation amount, based on 100 parts by weight of the base particle, is preferably 30 parts by weight or less, more preferably 20 parts by weight or less, especially preferably 15 parts by weights or less, from the viewpoint of the dissolubility.

[0022]

4. Fine Powder

In the present specification, the term "fine powder" is a powder for coating the surface of a uni-core detergent particle which is formulated for the purpose of improving the flowability of the detergent particle, and those having high ion exchanging ability and high alkalizing ability are preferable from the viewpoint of the detergency. Concretely, aluminosilicates are preferable. Aside from the aluminosilicates, inorganic fine powders of calcium silicates, silicon dioxide, bentonite, talc, clay, amorphous silica derivatives and

silicate compounds such as crystalline silicate compounds are preferable. In addition, metal soaps of which primary particle has a size of 10 μm or less can be similarly used.

[0023]

The fine powder of which primary particle has an average particle size of from 0.1 to 10 μm is preferable, from the viewpoints of an improvement in the coating ratio of the surface of the uni-core detergent particle and an improvement in the flowability of the detergent particle. The average particle size of the fine powder can be measured by a method utilizing light scattering, for instance, by a particle analyzer (commercially available by Horiba, LTD.), or it may be measured by a microscopic observation.

[0024]

The amount of the fine powder used, based on 100 parts by weight of the mixture obtainable in step (A), is 5 parts by weight or more, more preferably 10 parts by weight or more, from the viewpoint of obtaining a uni-core particle. In addition, the amount is 100 parts by weight or less, preferably 75 parts by weight or less, especially preferably 50 parts by weight or less, from the viewpoint of the flowability.

[0025]

5. Process for Preparing Uni-Core Detergent Particle

5-1. Step (A)

As to the mixing conditions in step (A), there may be selected mixing conditions such that the base particle does not substantially undergo breakdown. For instance, when a mixer comprising agitation impellers is used, the agitation impellers have a Froude number of preferably 8 or less, more preferably from 4 or less, especially preferably from 2 or less, from the viewpoint of suppression of the breakdown of the base particle. The agitation impellers have a Froude number of preferably from 0.5 or more, more preferably from 0.8 or more.

[0026]

Further, there may be also employed a mixer comprising agitation impellers and disintegration impellers. When powders and liquids are mixed by using the above mixer, the disintegration impellers have been conventionally subjected to high-speed rotation, from the viewpoint of accelerating mixing. However, in the present invention, in the above embodiment, it is preferable not to substantially rotate the disintegration impellers, from the viewpoint of the suppression of breakdown of the base particle. The phrase "substantially not rotate the disintegration impellers" refers to completely no rotations of the disintegration impellers at all, or some rotations of the disintegration impellers, for the purpose

of preventing residence of various raw materials near the disintegration impellers, within a range such that the base particle does not substantially undergo breakdown, in consideration of shapes, sizes, and the like of the disintegration impellers. Concretely, in a case where the disintegration impellers are continuously rotated, the Froude number is 200 or less, preferably 100 or less, and in a case where the disintegration impellers are intermittently rotated, the Froude number is not particularly limited. The mixture can be obtained without substantially undergoing breakdown of the base particle by mixing under the conditions described above. In the present specification, a state where a "base particle does not substantially undergo breakdown" refers to a state such that 70% or more of the base particle in the mixture maintains its shape. Its method for confirmation includes, for instance, a method of subjecting to observation granules obtained after extracting a soluble fraction from a mixture obtained by using an organic solvent.

[0027]

The Froude number as defined in the present specification is calculated by the following equation.

Froude Number = V^2 / (R x g),

wherein V: a peripheral speed [m/s] of a tip end portion of an agitating impeller or disintegration

impeller;

R: a rotational radius [m] of an agitating impeller or disintegration impeller; and

g: gravitational acceleration [m/s^2].

[0028]

As the mixer temperature during mixing, a temperature at which the surfactant composition and base particles can be efficiently mixed with suppressing the breakdown of the base particle is preferable. For instance, the temperature of the mixed components is preferably equal to or higher than the pour point, more preferably higher than the pour point by 10°C or more, still more preferably higher than the pour point by 20°C or more. The mixing time is preferably from 2 to 10 minutes or so. The adjustment of the mixer temperature can be carried out by allowing to flow cold water or warm water through a jacket or the like. Therefore, the device used for mixing is preferably one having a structure equipped with a jacket. The pour point of the surfactant composition is a value determined by a method in accordance with JIS K 2269.

[0029]

A process for mixing a surfactant composition and base particle may be a batch process or a continuous process. In the case of mixing by a batch process, it is preferable to previously supply base particle in a mixer,

and thereafter add thereto a surfactant composition in a liquid state. Especially, it is preferable to feed the surfactant composition in a liquid state by spraying. The temperature of the surfactant composition to be fed is preferably higher than a pour point of the surfactant composition by 10°C or more, more preferably higher than the pour point by 20°C or more.

[0030]

In the case where mixing is carried out by a batch process, the mixer is not particularly limited, as long as a mixer which can satisfy the present invention is employed. Examples of the mixers include (1) a mixer in which blending of powders is carried out by having an agitating shaft in the inner portion of a mixing vessel and attaching agitating impellers on the agitating shaft, including Henschel Mixer (manufactured by Mitsui Miike Machinery Co., Ltd.), High-Speed Mixer (Fukae Powtec Corp.), Vertical Granulator (manufactured by Powrex Corp.), Lödige Mixer (manufactured by Matsuzaka Giken Co., Ltd.), PLOUGH SHARE Mixer (manufactured by PACIFIC MACHINERY & ENGINEERING Co., LTD.), and the like; (2) a mixer in which blending is carried out by rotating spiral ribbon impellers in a non-rotatable vessel which is cylindrical, semi-cylindrical, or conical, including Ribbon Mixer (manufactured by Nichiwa Kikai Kogyo K.K.), Batch Kneader

(manufactured by Satake Kagaku Kikai Kogyo K.K.), and the like; (3) a mixer in which blending is carried out by revolving a screw along a conical vessel, with autorotation centering about a rotating shaft arranged parallel to the vessel wall, including Nauta Mixer (manufactured by Hosokawa Micron Corp.), and the like.

Among the above mixers, particular preference is given to a mixer in which blending of powders is carried out by having an agitating shaft arranged along the center line of a horizontal, cylindrical blending vessel and attaching agitating impellers arranged on the agitating shaft, including Lödige Mixer (manufactured by Matsuzaka Giken Co., Ltd.), PLOUGH SHARE Mixer (manufactured by PACIFIC MACHINERY & ENGINEERING Co., LTD.), and the like.

[0031]

In addition, in a case where mixing is carried out in a continuous process, the mixer is not particularly limited, as long as a continuous mixer which can satisfy the present invention is employed. For instance, a base particle and a surfactant composition may be mixed by using a continuous mixer among the above mixers.

[0032]

The form of the mixture of the powder and the liquid is described in literatures such as "*Funtaikogaku Yogo Jiten*" (published by Nikkan Kogyo Shinbunsha, 1981), which

is summarized in Table 1. It is more preferable that the mixture obtainable in step (A) has any one of forms in Funicular Region II, Capillary Region, and Slurry Region. Such a form of the mixture means that the surfactant composition in the mixture is present in an amount capable of supporting the base particle or more. By having a mixture in such a form, the surfactant composition can be formulated at a high level, as compared to those in Pendular Region and Funicular Region I. Moreover, the mixture can have a whipping form, and as a result, a shearing force (kneading resistance) acting among the base particles can be reduced. Therefore, the breakdown of the base particle can be suppressed. In addition, the effects of surface coating by fine powder can be efficiently exhibited, as long as the mixture has any one of forms in Funicular Region II, Capillary Region, and Slurry Region, so that a uni-core detergent particle having excellent flowability can be obtained. The confirmation of which form of the region the mixture belongs can be carried out by using a magnifying glass or the like.

[0033]

[Table 1]

Table 1

	Form of Mixture				
	Pendular Region	Funicular Region I	Funicular Region II	Capillary Region	Slurry Region
Solid	Conti-nuous	Conti-nuous	Conti-nuous	Discon-tinuous	Discon-tinuous
Liquid	Discon-tinuous	Conti-nuous	Conti-nuous	Conti-nuous	Conti-nuous
Gas	Conti-nuous	Conti-nuous	Discon-tinuous	None	None

[0034]

In order that the mixture has any one of forms of Funicular Region II, Capillary Region and Slurry Region, the amount of the surfactant composition may be appropriately adjusted in consideration of the amount capable of being supported to the base particle.

[0035]

In addition, when the powder raw materials other than the base particle are formulated in step (A), it is preferable that the powder raw materials are supplied to the mixer before adding the surfactant composition. It is preferable that the mixing conditions when the powder raw materials are formulated are the same conditions as those where the base particle and the surfactant composition are

mixed.

[0036]

5-2. Step (B)

In this step, the fine powder is mixed with the mixture obtainable in step (A), whereby the fine powder coats the surface of the mixture (a base particle comprising a surfactant composition), to give a uni-core detergent particle having excellent flowability. When the surfactant composition forms a continuous phase as in a case where the mixture has a form of Funicular Region II, Capillary Region and Slurry Region, the fine powder has the functions of the pulverization aid for making the continuous phase discrete in the early stage of mixing.

[0037]

As the mixer temperature during mixing, a temperature at which coating with the fine powder can be efficiently carried out with suppressing the breakdown of the base particle is preferable. For instance, the temperature of the components is preferably higher than the pour point of the added surfactant composition in step (A) by 10°C or more, more preferably higher than the pour point by 20°C or more. In addition, from the viewpoint of the thermal stability of a surfactant composition, the mixer temperature is preferably 95°C or lower, more preferably 90°C or lower. The mixing time is preferably from 0.5 to

3 minutes or so.

[0038]

As the mixing conditions in step (B), there may be selected mixing conditions such that the form of the base particle comprising the surfactant composition is substantially maintained. Preferable mixing conditions are the use of a mixer comprising both agitation impellers and disintegration impellers. When the above mixer is used, the agitation impellers provided in the mixer have a Froude number of preferably 10 or less, more preferably 7 or less, from the viewpoint of the suppression of breakdown of the base particle. The Froude number is preferably 2 or more, more preferably 3 or more, from the viewpoints of the efficiencies in the mixing with the fine powder and in the dispersion of the fine powder. The disintegration impellers have a Froude number of preferably 200 or more, more preferably 500 or more, from the viewpoints of the efficiencies in the mixing with the fine powder and in the dispersion of the fine powder. The Froude number is preferably 8000 or less, more preferably 5000 or less, from the viewpoint of the suppression of the breakdown of the base particle. If the Froude number is in this range, the uni-core detergent particle having excellent flowability can be obtained. In the present specification, the phrase "the base particle comprising

the surfactant composition, the form of which is substantially maintained" means that 70% or more of each of the resulting detergent particle is constituted by one base particle, and that the base particle does not undergo breakdown. As the method of its confirmation, the same means as those of step (A) can be employed.

[0039]

Preferable mixers include, among the mixers exemplified in step (A), those comprising both agitation impellers and disintegration impellers. When the mixers as described above are used, it is preferable from the viewpoint of simplification of equipments because the same device can be used in step (A) and step (B). The devices as described above include Lödige Mixer (manufactured by Matsuzaka Giken Co., Ltd.), PLOUGH SHARE Mixer (manufactured by PACIFIC MACHINERY & ENGINEERING Co., LTD.), and the like.

[0040]

6. Uni-Core Detergent Particle

The uni-core detergent particle obtainable by the process of the present invention includes a detergent particle comprising a base particle as a core, wherein a detergent particle substantially comprises one base particle as a core in one detergent particle.

[0041]

As an index for expressing uni-core property of the detergent particle, the degree of particle growth defined by the following equation can be used. The uni-core detergent particle in the present invention have a degree of particle growth of 1.5 or less, preferably 1.3 or less.

$$\text{Degree of Particle Growth} = \frac{\text{Average Particle Size of Final Detergent Particle}}{\text{Average Particle Size of Base Particle}}$$

The term "final detergent particle" refers to a detergent particle obtained after step (B).

[0042]

In the above uni-core detergent particle, since the intraparticle aggregation is suppressed, there is an advantage that a desired detergent is obtained in a high yield without forming particles (aggregated particle) having sizes outside the desired particle size range.

[0043]

When the base particle is a spray-dried particle, the uni-core detergent particle obtained by the process of the present invention can realize fast dissolubility. The term "fast dissolubility" refers to a property wherein the dissolution rate of the detergent particle as calculated by the following method is 90% or more.

[0044]

A 1-L beaker (a cylindrical form having an inner diameter of 105 mm and a height of 150 mm, for instance, a 1-L glass beaker manufactured by Iwaki Glass Co., Ltd.) is charged with 1 L of hard water cooled to 5°C and having a water hardness corresponding to 71.2 mg $CaCO_3$/L (a molar ratio of Ca/Mg: 7/3). With keeping the water temperature constant at 5°C with a water bath, water is stirred with a stirring bar (35 mm in length and 8 mm in diameter, for instance, Model "TEFLON MARUGATA-HOSOGATA" manufactured by ADVANTEC) at a rotational speed (800 rpm), such that a depth of swirling to the water depth is about 1/3. The detergent particle is weighed so as to be 1.00 g is supplied and dispersed in hard water mentioned above with stirring, and stirring is continued. After 60 seconds from supplying the detergent particle, a liquid dispersion of the detergent particle in the beaker is filtered with a standard sieve (100 mm in diameter) and a sieve-opening of 74 μm as defined by JIS Z 8801 (corresponding to ASTM No. 200) of a known weight. Thereafter, water-containing detergent particle remaining on the sieve is collected in an open vessel of a known weight together with the sieve. Incidentally, the operation time from the start of filtration to collection of the sieve is set at 10 ± 2 sec. The insoluble remnants of the collected detergent particle are dried for one hour in an electric dryer heated to 105°C.

Thereafter, the dried insoluble remnants are cooled by keeping in a desiccator with a silica gel at 25°C for 30 minutes. After cooling the insoluble remnants, a total weight of the dried insoluble remnants of the detergent, the sieve and the collecting vessel is measured, and the dry weight of the detergent particle remaining on the sieve is determined. Thereafter, the dissolution rate (%) of the detergent particle is calculated by the following equation. Incidentally, the weight is determined by using an accurate balance.

[0045]

$$\text{Dissolution Rate (\%)} = \{1 - (T/S)\} \times 100$$

wherein S is a weight (g) of the detergent particle supplied; and

T is a dry weight (g) of the detergent particle remaining on the sieve.

[0046]

The bulk density of the uni-core detergent particle is 500 g/L or more, preferably from 500 to 1,000 g/L, more preferably from 600 to 1,000 g/L, especially preferably from 650 to 850 g/L. The method for determining a bulk density is the same as that for the base particle.

[0047]

The average particle size of the uni-core detergent particle is preferably from 150 to 500 μm, more preferably

from 180 to 350 µm. The method for determining an average particle size is the same as that for the base particle.

[0048]

The flowability of the uni-core detergent particle is evaluated as flow time of preferably 10 seconds or shorter, more preferably 8 seconds or shorter. The flow time is a time period required for dropping 100 mL of powder from a hopper used in the determination of the bulk density as defined in JIS K 3362.

[0049]

The yield of the uni-core detergent particle is calculated from a weight percentage of a sample passing through a sieve having a sieve-opening of 1000 µm when the average particle size is determined. The yield is preferably 90% or more, more preferably 95% or more.

[0050]

[Examples]

Example 1

A detergent particle was obtained according to the following process.

One-hundred parts by weight (20 kg) of a base particle as listed in Table 2 were supplied into Lödige Mixer (commercially available from Matsuzaka Giken Co., Ltd.; capacity: 130 L; equipped with a jacket), and the rotation of a main shaft (equipped with agitation

impellers; rotational speed of the main shaft: 60 rpm; Froude number of agitation impellers: 1) was started. Incidentally, hot water at 80°C was allowed to flow into the jacket at 10 L/minute, without rotating a chopper (equipped with disintegration impellers). Fifty parts by weight (10 kg) of a liquid surfactant composition at 80°C was supplied into the above mixer over a period of 2 minutes, and thereafter the components were mixed for 5 minutes. Subsequently, 15 parts by weight (3 kg) of fine powder was supplied into this Lödige Mixer. The main shaft (rotational speed of the main shaft: 120 rpm; Froude number of agitation impellers: 4) and the chopper (rotational speed of the chopper: 3600 rpm; Froude number of disintegration impellers: 1300) were rotated for 1 minute, and thereafter 28 kg of a detergent particle was obtained. The composition and the properties of the obtained detergent particle are shown in Table 2.

[0051]

[Table 2]

Table 2

	Examples						
	1	2	3	4	5	6	7
Composition (Parts by Weight)							
Surfactant Composition							
Surfactant Composition 1	-	-	-	-	-	50	-
Surfactant Composition 2	50	50	30	70	55	-	30
Base Particles							
Spray-Dried Particle	100	100	100	100	100	100	50
Sodium Carbonate *1)	-	-	-	-	-	-	50
Powdery Raw Material							
Sodium Carbonate *2)	-	-	-	-	5	-	-
Crystalline Silicate *3)	-	-	-	-	5	-	-
Fine Powder							
Crystalline Aluminosilicate *4)	-	50	-	-	50	50	-
Amorphous Aluminosilicate *5)	15	-	10	30	-	-	15
Properties, etc.							
Average Particle Size [μm]	245	260	235	290	250	230	275
Degree of Particle Growth	1.09	1.16	1.04	1.29	1.11	1.02	1.07
Yield [%]	98.6	98.5	99.4	96.8	98.2	98.9	98.8
Bulk Density [g/L]	710	770	640	690	780	780	880
Flowability [s]	6.4	6.5	6.6	5.9	6.1	6.0	6.2
Dissolution Ratio [%]	93	-	-	-	-	-	-

- Continued -

- Continued -

	Comparative Examples		
	1	2	3
Composition (Parts by Weight)			
Surfactant Composition			
Surfactant Composition 1	-	-	30
Surfactant Composition 2	50	50	-
Base Particle			
Spray-Dried Particle	100	100	100
Sodium Carbonate *1)	-	-	-
Powdery Raw Material			
Sodium Carbonate *2)	-	-	-
Crystalline Silicate *3)	-	-	-
Fine Powder			
Crystalline Aluminosilicate *4)	-	-	-
Amorphous Aluminosilicate *5)	15	15	-
Properties, etc.			
Average Particle Size [μm]	415	380	Undeterminable
Degree of Particle Growth	1.84	1.69	Undeterminable
Yield [%]	84.3	78.5	Undeterminable
Bulk Density [g/L]	780	720	Undeterminable
Flowability [s]	5.8	11.5	Undeterminable
Dissolution Ratio [%]	65	72	-

*1): "DENSE ASH" (commercially available from Central Glass Co., Ltd.), average particle size of 290 μm, bulk density of 980 g/L, and particle strength of 2300 kg/cm^2

*2): "LIGHT ASH" (commercially available from Central Glass Co., Ltd.), average particle size of 100 μm

*3): Product prepared by pulverizing Na-SKS-6 (δ-$Na_2Si_2O_5$) (commercially available from Clariant) to an average particle size of 8 μm

*4): Zeolite 4A-type, average particle size: 3.5 μm

*5): Product prepared by pulverizing the composition of Preparation Example 2 described in Japanese Patent Laid-Open No. Hei 9-132794 to an average particle size of 8 μm

[0052]

Examples 2 to 7

A detergent particle was obtained in the same manner as in Example 1 with each of the compositions listed in Table 2. The properties of the obtained detergent particle are shown in Table 2. Incidentally, in Example 5, the powdery raw materials were supplied simultaneously with the base particle.

[0053]

Regarding to the form of the mixture prior to mixing with fine powder, as a result of determining from the observation with a magnifying glass, the form of the mixture of Example 3 was in Pendular Region, the forms of

the mixtures of Examples 1, 2, and 5 to 7 were in Funicular II region, and the form of the mixture of Example 4 was in Capillary Region. The detergent particles of Examples 4 and 5 were more excellent in the detergency than the detergent particle of Example 3. In addition, the detergent particle of Examples 1 had fast dissolubility. In addition, the detergent particles of Examples 1 to 5, and 7 were more excellent in the exudation preventing property for surfactant composition than the detergent particle of Example 6.

It was found from the data on the degree of particle growth that each of the detergent particles obtained in Examples 1 to 7 was uni-core detergent particle.

[0054]

In addition, soluble matter was extracted and removed from the obtained detergent particle using an organic solvent, and the resulting particle were observed. As a result, in each of Examples, the base particle did not substantially undergo breakdown, and the forms of the base particle comprising the surfactant composition were substantially maintained.

[0055]

Incidentally, the surfactant compositions and the spray-dried particle used were the following:

Surfactant Composition 1: polyoxyethylene alkyl ether

[commercially available from Kao Corporation under the trade name: "EMULGEN 108 KM" (average moles of ethylene oxides: 8.5; number of carbon atoms in alkyl moiety: 12 to 14; and melting point: 18°C)]

Surfactant Composition 2: a composition of polyoxyethylene alkyl ether/polyethylene glycol/ LAS-Na/water = 42/8/42/8 (weight ratio) (pour point being 45°C)

polyoxyethylene alkyl ether ("EMULGEN 108 KM")

polyethylene glycol [commercially available from Kao Corporation under the trade name: "K-PEG 6000" (average molecular weight: 8500; melting point: 60°C)]

LAS-Na: dodecylbenzenesulfonate (commercially available from Kao Corporation under the trade name: NEOPELEX FS).

Spray-Dried Particle: a bulk density of 620 g/L, an average particle size of 225 μm, a particle strength of 320 kg/cm^2, and a composition: zeolite/sodium polyacrylate/sodium carbonate/sodium sulfate/water = 50/10/20/15/5 (weight ratio).

[0056]

The spray-dried particle used herein was prepared as follows.

Four-hundred and eighty kilograms of water was supplied to a 1-m^3 mixing vessel comprising agitation

impellers. After the water temperature reached 55°C, 150 kg of an aqueous solution of 40% by weight sodium polyacrylate was added thereto. The mixture was stirred for 15 minutes, and thereafter 120 kg of sodium carbonate and 90 kg of sodium sulfate were added thereto. The resulting mixture was stirred for additional 15 minutes, and thereafter 300 kg of zeolite was added thereto. The resulting mixture was stirred for 30 minutes, to give a homogenous slurry. The final temperature of this slurry was 58°C.

[0057]

This slurry was fed to a spray-drying tower by a pump, and sprayed from a pressure-spray nozzle attached near the top of the tower at a spraying-pressure of 25 kg/cm^2. The high-temperature gas to be supplied to the spray-drying tower was supplied from the bottom of the tower at a temperature of 225°C, and discharged from the top of the tower at 105°C.

[0058]

Comparative Example 1

A detergent particle was obtained according to the following process.

One-hundred parts by weight (20 kg) of a base particle was supplied into Lödige Mixer (commercially available from Matsuzaka Giken Co., Ltd.; capacity: 130 L;

equipped with a jacket), and the rotation of a main shaft (equipped with agitation impellers; rotational speed of the main shaft: 120 rpm; Froude number of agitation impellers: 4) and a chopper (equipped with disintegration impellers; rotational speed of the chopper: 3600 rpm; Froude number of disintegration impellers: 1300) was started. Incidentally, hot water at 80°C was allowed to flow into the jacket at 10 L/minute. Fifty parts by weight (10 kg) of a liquid surfactant composition at 80°C was supplied into the mixer over a period of 2 minutes, and thereafter the components were mixed for 5 minutes. The form of this mixture was in Funicular I region.

[0059]

Subsequently, 15 parts by weight (3 kg) of fine powder was supplied into this Lödige Mixer, and the main shaft (rotational speed of the main shaft: 120 rpm; Froude number of agitation impellers: 4) and the chopper (rotational speed of the chopper: 3600 rpm; Froude number of disintegration impellers: 1300) were rotated for 1 minute. Thereafter, 28 kg of detergent particle was obtained. The composition and the properties of the obtained detergent particle are shown in Table 2.

From the data on the degree of particle growth, the obtained detergent particle was not a uni-core detergent particle. In addition, the yield was poor. In addition,

this detergent particle was poor in the dissolubility, as compared to that of Example 1 having the same composition.

[0060]

Comparative Example 2

A detergent particle was obtained according to the following process.

One-hundred parts by weight (20 kg) of base particle was supplied into Lödige Mixer (commercially available from Matsuzaka Giken Co., Ltd.; capacity: 130 L; equipped with a jacket), and the rotation of a main shaft (equipped with agitation impellers; rotational speed of the main shaft: 60 rpm; Froude number of agitation impellers: 1) was started. Incidentally, hot water at 80°C was allowed to flow into the jacket at 10 L/minute, without rotating a chopper (equipped with disintegration impellers). Fifty parts by weight (10 kg) of the above liquid surfactant composition at 80°C was supplied into the mixer over a period of 2 minutes, and thereafter the components were mixed for 5 minutes. The form of this mixture was in Funicular II region.

[0061]

Subsequently, 15 parts by weight (3 kg) of fine powder was supplied into this Lödige Mixer, and the main shaft (rotational speed of the main shaft: 60 rpm; Froude number of agitation impellers: 1) was rotated for 1 minute.

Thereafter, 28 kg of detergent particle was obtained. The chopper was not also rotated in this process. The composition and the properties of the obtained detergent particle are shown in Table 2.

From the data on the degree of particle growth, the obtained detergent particle was not a uni-core detergent particle. In addition, the yield was poor. In addition, the flowability and the dissolubility of this detergent particle were also poor.

[0062]

Comparative Example 3

A detergent particle was obtained in the same manner as in Example 1 with the composition listed in Table 2, provided that the mixing process for the fine powder was not carried out. The obtained detergent particle was not in a powdery state (Pendular region), so that the value of each of the properties could not be determined.

The obtained detergent particle had a low bulk density, and the properties were so poor in texture that the flowability was undeterminable.

[0063]

[Effects of the Invention]

According to the present invention, there can be provided a process for preparing a uni-core detergent particle in which the preparation steps can be simplified,

the variations in the properties of the detergent particle against the variations in the formulated amount of the surfactant composition can be suppressed, further the flowability of the detergent particle is excellent, and the surfactant composition can be formulated in large amounts.

[Document] Abstract

[Abstract]

[Problems]

To provide in a process for preparing a uni-core detergent particle comprising a surfactant composition, the process for obtaining the uni-core detergent particle in high yield capable of easily adjusting an average particle size and a particle size distribution by selection of a base particle in a simple preparation process, wherein the variations of the average particle size and the particle size distribution of the detergent particle are small with respect to the variation of the amount of the surfactant composition formulated.

[Solving Means]

A process for preparing detergent particle, comprising the steps of (A): mixing a base particle and 20 to 100 parts by weight of a surfactant composition, based on 100 parts by weight of the base particle, the base particle having an average particle size of from 150 to 500 µm, a bulk density of 400 g/L or more, and a particle strength of 50 kg/cm^2 or more, under mixing conditions such that the base particle does not substantially undergo breakdown, to give a mixture; and (B): mixing the mixture obtained in step (A) with 5 to 100 parts by weight of fine powder, based on 100 parts by weight of the mixture, with

substantially maintaining the shape of the base particle containing the surfactant composition, to give a uni-core detergent particle,

wherein the uni-core detergent particle has a degree of particle growth of 1.5 or less, and a bulk density of 500 g/L or more.

[Selected Drawing] none